



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Office of Research and Development
Washington, D.C. 20460



ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM VERIFICATION STATEMENT

TECHNOLOGY TYPE:	POLYCHLORINATED BIPHENYL (PCB) FIELD ANALYTICAL TECHNIQUES
APPLICATION:	MEASUREMENT OF PCBs IN SOILS AND SOLVENT EXTRACTS
TECHNOLOGY NAME:	L2000 PCB/CHLORIDE ANALYZER
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The U.S. Environmental Protection Agency (EPA) has created a program to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the Environmental Technology Verification (ETV) Program is to further environmental protection by substantially accelerating the acceptance and use of improved and more cost effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. This document summarizes the results of a demonstration of the Dexsil L2000 PCB/Chloride Analyzer.

PROGRAM OPERATION

EPA, in partnership with recognized testing organizations, objectively and systematically evaluates the performance of innovative technologies. Together, with the full participation of the technology developer, they develop plans, conduct tests, collect and analyze data, and report findings. The evaluations are conducted according to a rigorous demonstration plan and established protocols for quality assurance. EPA's National Exposure Research Laboratory, which conducts demonstrations of field characterization and monitoring technologies, with the support of the U.S. Department of Energy's (DOE's) Environmental Management (EM) program, selected Oak Ridge National Laboratory as the testing organization for the performance verification of polychlorinated biphenyl (PCB) field analytical techniques.

DEMONSTRATION DESCRIPTION

In July 1997, the performance of six PCB field analytical techniques was determined under field conditions. Each technology was independently evaluated by comparing field analysis results to those obtained using approved reference methods. Performance evaluation (PE) samples were also used to assess independently the accuracy and comparability of each technology.

The demonstration was designed to detect and measure PCBs in soil and solvent extracts. The demonstration was conducted at the Oak Ridge National Laboratory (ORNL) in Oak Ridge, Tennessee, from July 22 through July 29, 1997. The study was conducted under two environmental conditions. The first site was outdoors, with naturally fluctuating temperatures and relative humidity conditions. The second site was inside a controlled environmental chamber, with generally cooler temperatures and lower relative humidities. Multiple soil types, collected from sites in Ohio, Kentucky, and Tennessee, were analyzed in this study. Solutions of PCBs were also analyzed to simulate extracted surface wipe

samples. The results of the soil and extract analyses conducted under field conditions by the technology were compared with results from analyses of homogeneous replicate samples conducted by conventional EPA SW-846 methodology in an approved reference laboratory. Details of the demonstration, including a data summary and discussion of results, may be found in the report entitled *Environmental Technology Verification Report: Electrochemical Technique/Ion Specific Electrode, Dexsil Corporation, L2000 PCB/Chloride Analyzer*, EPA/600/R-98/109.

TECHNOLOGY DESCRIPTION

The L2000 PCB/Chloride Analyzer (dimensions: 8" × 8" × 4.5") is a field-portable instrument, weighing approximately 3.5 lb, designed to quantify PCB concentration in soils, dielectric fluids, and surface wipes. Sample preparation consists of extraction and dehalogenation of the PCB. A 10-g sample of soil is weighed into a polyethylene test tube. The soil is extracted with a nonchlorinated solvent from a premeasured ampule. (Note that a newly developed hydrocarbon solvent system was used for the demonstration analyses.) The soil is allowed to settle, and the supernatant is decanted onto a Florisil column. The solution is passed through the column, where all of the water and inorganic chloride is removed. Five milliliters of the solution are collected in a polyethylene reaction tube. Two glass ampules contained in the reaction tube are broken, introducing metallic sodium to the extract solution. The sodium strips the covalently bound chlorine atoms off the PCB molecule. The mixture is then shaken for 10 s and allowed to react for a total of 1 min. An aqueous extraction solution is added to the reaction tube to adjust the pH, destroy the excess sodium, and extract and isolate the newly formed chloride ions in an aqueous buffered solution. The aqueous layer is decanted, filtered, and collected in an analysis vial. A chloride-ion-specific electrode is put into this aqueous solution to measure the millivolt potential of the chloride solution. The potential is then converted to a PCB concentration in terms of parts per million (ppm).

VERIFICATION OF PERFORMANCE

The following performance characteristics of the L2000 PCB/Chloride Analyzer were observed:

Detection limits: EPA defines the method detection limit (MDL) as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL was calculated to be 7.1 ppm based on the performance evaluation sample analyses. By use of a line fitted to a plot of the L2000-measured PCB concentrations versus the certified PE values, bias in the L2000 data can be corrected. After compensation for bias, the resulting L2000 MDL agrees with Dexsil's specified MDL of 2 ppm.

Throughput: Throughput was 5 samples/hour under the outdoor conditions and 10 samples/hour under the chamber conditions. This rate included sample preparation and analysis.

Ease of Use: Two operators analyzed samples during the demonstration, but the technology can be run by a single trained operator. Minimal training (<1 hour) is required to operate the L2000, provided the user has a fundamental understanding of basic chemical techniques.

Completeness: The L2000 generated results for all 232 PCB samples for a completeness of 100%.

Blank results: PCBs were detected above the L2000's MDL for four of the eight blank samples. Therefore, the percentage of false positive results was 50%. These results were obtained for both soil and extract samples. The L2000 reported no false negative results.

Precision: The overall precision, based on average relative standard deviations (RSDs), was 23% for soil samples and 14% for extract samples. The L2000's precision was comparable to that of the reference laboratory (21% RSD for soils and 14% RSD for extracts). At higher concentrations (>125 ppm), the L2000 was more precise than the reference laboratory (4% versus 19% RSD).

Accuracy: Accuracy was assessed using PE soil and extract samples. The data showed that the L2000 exhibited a significantly high bias. The overall accuracy, based on average percent recoveries, was 208% for PE soil samples and 149% for extract samples. Evaluation of the data generated at each site indicated that there were no significant differences between the two data sets based on environmental conditions.

Comparability: This demonstration showed that the L2000 generated data that exhibited a linear correlation to the reference laboratory data. The coefficient of determination (R^2), which is a measure of the degree of correlation between the reference laboratory and the L2000 data, was 0.854 when all soil samples (0 to 700 ppm) were considered. For the concentration range from 0 to 125 ppm, the R^2 value was 0.781. Most of the percent difference values were greater than 100% when the L2000 results were compared directly with the reference laboratory results.

Regulatory decision-making: One objective of this demonstration was to assess the technology's ability to perform at regulatory decision-making levels for PCBs, specifically 50 ppm for soils and 100 $\mu\text{g}/100\text{cm}^2$ for surface wipes. For PE and environmental soil samples in the range of 40 to 60 ppm, the precision was high (12% RSD), but the measured concentrations were biased high (147% recovery). For extract samples representing surface wipe sample concentrations of 100 $\mu\text{g}/100\text{cm}^2$ and 1000 $\mu\text{g}/100\text{cm}^2$ (assuming a 1000 cm^2 wipe sample), measurements were precise (14% RSD), but indicated a high bias (149% recovery), especially for the lower concentrations.

Data quality levels: Because the PCB data generated in this demonstration strongly correlated with the reference laboratory results, it may be possible for Dexsil's L2000 PCB/Chloride Analyzer to be used quantitatively, but the high bias must be considered. The overall performance was characterized as consistently biased but precise.

The results of the demonstration show that the Dexsil L2000 PCB/Chloride Analyzer can provide useful, cost-effective data for environmental problem-solving and decision-making. Undoubtedly, it will be employed in a variety of applications, ranging from serving as a complement to data generated in a fixed analytical laboratory to generating data that will stand alone in the decision-making process. As with any technology selection, the user must determine if this technology is appropriate for the application and the project data quality objectives. For more information on this and other verified technologies, visit the ETV web site at <http://www.epa.gov/etv>.

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NOTICE: EPA verifications are based on an evaluation of technology performance under specific, predetermined criteria and the appropriate quality assurance procedures. EPA makes no expressed or implied warranties as to the performance of the technology and does not certify that a technology will always, under circumstances other than those tested, operate at the levels verified. The end user is solely responsible for complying with any and all applicable Federal, State, and Local requirements.

